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NASA TM X-73663

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(NASA-TM-X-73663) EFFECTS OF HEAT TREATING
PM RENE' 95 SLIGHTLY BELOW THE GAMMA' SOLVUS
(NASA) 19 p HC A02/MF A01 CSCI 11F

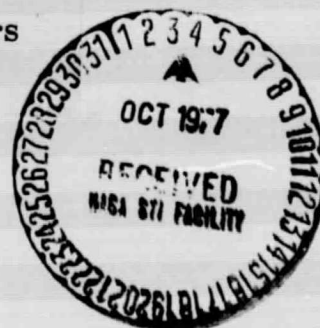
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**EFFECTS OF HEAT TREATING PM RENE' 95 SLIGHTLY
BELOW THE γ' SOLVUS**

by Robert L. Dreshfield
Lewis Research Center
Cleveland, Ohio 44135

TECHNICAL PAPER presented at the
One hundred sixth Annual Meeting of the American
Institute of Mining, Metallurgical, and Petroleum Engineers
Atlanta, Georgia, March 6-10, 1977



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SLIGHTLY BELOW THE γ' SOLVUS

by Robert L. Dreshfield

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

ABSTRACT

An investigation was performed on As-HIP Rene' 95 to obtain additional information on the variation of the amount of γ' with solutioning temperatures near the γ' solvus temperature and the resulting effects on tensile and stress rupture strengths of As-HIP Rene' 95. The amount of γ' phase was found to increase at a rate of about 0.5% per degree Celsius as the temperature decreased from the solvus temperature to about 50° below the γ' solvus temperature. The change in the amount of γ' phase with decreasing solutioning temperature was observed to be primarily associated with decreasing solubilities of Al+Ti+Nb and increasing solubility of Cr in the γ phase. For As-HIP Rene' 95 solutioned at either 1107 or 1135°C, and subsequently water quenched and double aged for 4 hours at 815°C followed by 24 hours at 650°C, the higher solution temperature resulted in significantly greater yield strengths at room temperature and 650°C and a greater room temperature ultimate strength. Also, longer stress rupture lives at 650°C were associated with the higher solution temperature.

STAR Category 26

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by Robert L. Dreshfield

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

SUMMARY

An investigation was performed on As-HIP Rene' 95 to obtain additional information on the variation of the amount of γ' with solutioning temperatures near the γ' solvus temperature and the resulting effects on tensile and stress rupture strengths of As-HIP Rene' 95. The amount of γ' phase was found to increase at a rate of about 0.5% per degree Celsius as the temperature decreased from the solvus temperature to about 50° below the γ' solvus temperature. The change in the amount of γ' phase with decreasing solutioning temperature was observed to be primarily associated with decreasing solubilities of Al+Ti+Nb and increasing solubility of Cr in the γ phase. For As-HIP Rene' 95 solutioned at either 1107 or 1135°C, and subsequently water quenched and double aged for 4 hours at 815°C followed by 24 hours at 650°C, the higher solution temperature resulted in significantly greater yield strengths at room temperature and 650°C and a greater room temperature ultimate strength. Also, longer stress rupture lives at 650°C were associated with the higher solution temperature.

INTRODUCTION

The more advanced Ni-base superalloys that are being considered for application in gas turbine disks are very difficult to forge and machine to their final shapes. To alleviate these problems and reduce the manufacturing cost of the component, there has been a considerable activity directed toward using as-hot-isostatically-pressed (As-HIP) superalloy powders for these highly stressed aircraft engine components. This approach eliminates the need for forging and reduces the amount of machining required to produce the required shapes.

The program reported in reference 1 has demonstrated that the high-strength disk alloy Rene' 95 can be processed by the As-HIP approach to achieve strength levels comparable to cast-and-wrought Rene' 95. To achieve these high strength levels, the As-HIP compacts were solution treated about 15°C below the γ' solvus temperature and rapidly cooled by oil or salt quenching prior to double aging at intermediate temperatures of 870 and 730°C.

The investigation reported here on As-HIP Rene' 95 was conducted to obtain additional information on the variation of the volume fraction of γ' with solution temperature near the γ' solvus. Also, a limited evaluation was made on the effects of varying solution temperatures on the resulting tensile and stress rupture strengths.

PROCEDURE

Materials

Materials from two sources were used in this study. The material used to determine the amount of γ' as a function of temperature was

provided by General Electric Company, Evendale, Ohio, and was produced from the -60 mesh powder supplied by Crucible Steel Co. as indicated in reference 1. It was received at NASA as a piece approximately 1 cm square x 5 cm long which was cut from a HIP piece used in the "Detailed Evaluation" phase of reference 1. It had been HIP'ed for 2 hours at 1120°C with a pressure of 103 MPa and heat treated by the preferred method of reference 1.

Because there was not sufficient material for mechanical property testing, the material used for the mechanical property testing was produced from -100 mesh argon atomized powder which was obtained directly from a commercial superalloy supplier. The composition of the powder was within the range shown in Table I. The powder was loaded in stainless steel cans approximately 13 cm long and 1.6 cm diameter. The cans were evacuated, baked, sealed, and then HIP'ed according to the same time/temperature/pressure parameters cited previously in reference 1.

Heat Treatment

To determine the volume fraction and composition of γ' as a function of solution temperature, the piece of As-HIP Rene' 95 provided by General Electric was first heated to 1190°C for two hours to dissolve the γ' and water quenched. (The γ' solvus temperature for this composition reported in reference 1 is 1165°C). Samples were then heated to 1077, 1113, 1138, 1154, and 1171°C for 6 hours and water quenched to room temperature. The solution temperatures were controlled to about 5°C.

The As-HIP cylinders from which mechanical test specimens were prepared were solution treated for 2 hours at either 1107 or 1135°C (1135°C being the recommended temperature in reference 1) and water

quenched to room temperature. Water quenching was used here in contrast to the slower oil and salt quenching in reference 1, to produce the greatest strength levels. It was thought that this would amplify the strength changes being evaluated - as will be shown later, this was not the case. The test cylinders were double aged sequentially for 4 hours at 815°C and 24 hours at 650°C with air cooling to room temperature after each thermal treatment.

Mechanical Testing

Duplicate tensile tests were run at room temperature and 650°C in air. Duplicate stress rupture tests were run in air at 650°C with a stress of 1035 MPa. For both tensile and stress rupture tests rod-type specimens having a cylindrical test section approximately 0.44 cm diameter were used. The tests were performed in accordance with ASTM recommended practices E8, E21, and E139.

Metallography

Metallographic specimens were prepared using conventional mechanical grinding and polishing procedures. For light microscopy, an etch consisting of 33 parts water, 33 parts nitric acid, 33 parts acetic acid, and 1 part hydrofluoric acid was used. For electron microscopy, an etch of 45 parts acetic acid, 40 parts lactic acid, 25 parts hydrochloric acid, and 10 parts nitric acid was used.

The amount of γ' resulting from solutioning at 1077, 1113, 1138, 1154, or 1171°C was determined metallographically from electron micrographs of surface replicas. Typical electron micrographs are shown in Figure 1. The structure can be seen to consist primarily of coarse-undissolved- γ' , fine-cooling and aging- γ' and a few carbides. The amount of coarse γ'

was determined by area measurement of several micrographs and is taken to be the volume fraction of γ' at the temperature of interest.

The metallographic technique was used to measure the amount of γ' because extractions (using the method of reference 2) contained from 34 to 40 weight percent residues, even for the sample quenched from above the γ' solvus temperature. Thus, it is apparent that water quenching cannot completely suppress γ' precipitation in Rene' 95.

The composition of γ' was determined by performing arc-emission spectroscopy on residues obtained by using an electrolyte of 1% ammonium sulfate, 1% citric acid in water (ref. 2). This composition was assumed to be the γ' composition. The γ composition was calculated by mass balance using the Rene' 95 composition from reference 1, the arc-emission spectrographically measured γ' composition and the volume fraction of γ' . No attempt was made to correct the volume fraction for density nor to account for carbides or borides likely to be included in the residuals.

RESULTS

Amount of γ' as Function of Temperature

The variation of amount of γ' with varying solution temperature is shown in figure 2. (The amount of γ' at 650°C in Figure 2 was determined by extraction from double aged material and the value of 54 weight percent was not corrected for carbide content or density. The γ' solvus temperature determined here is 1160°C which is within 5°C of that reported in reference 1. Differential thermal analysis was also run on this composition and a γ' solvus temperature of 1146°C was found (on cooling). These values for the γ' solvus temperature are considered to be in good agreement. Three independent techniques were used. The DTA method was subject

to cooling hysteresis while the metallographic methods used in this investigation and in reference 1 were subject to heating hysteresis.

At temperatures greater than about 1110°C, the curve in figure 2 is nearly linear. The rate of increase of amount of γ' with decreasing solution temperature is approximately 0.5 volume percent per degree Celcius from the solvus temperature about 1110°C. At the nominally specified solution temperature of 17°C below the γ' solvus temperature, the alloy has about 7.5% γ' . If one were to allow an 8°C variation about the specified solution temperature, the amount of undissolved γ' could vary from about 3% at 8°C below the solvus temperature to about 13% at 25°C below the solvus temperature. (If one compares figure 2 with the similar figure for Udimet 700 in reference 3, the similarity in shape is striking.)

An alternate way to view the data is to consider the relative amount of fine-cooling and aging γ' which would be available for maximum strengthening. If the solution temperature were to vary 17°C about the specified temperature, as little as 75 percent of the γ' may be available for strengthening at the low temperature limit compared to 95 percent at the high temperature limit. The relative amount of γ' available for strengthening increases at a rate of about 1% per degree Celcius as the temperature decreases from the solvus temperature. This variation would be expected to have an effect on the resulting mechanical properties.

Effect of Temperatures on Compositions of γ and γ'

The change in composition of γ and γ' with temperature was studied

using residues extracted from the same samples which were used to determine the volume fraction of γ' . (In this section of the report all compositions will be reported in atomic per cent.) While the compositions of the phases will refer to the solution temperature, the reader is reminded that the samples included γ' formed on cooling which is likely to be of a different composition from that existing at the solution temperature. The results are shown as a constitution diagram in Figure 3. The amount of γ' formers (Al+Ti+Nb) in the γ' phase is nearly independent of the solution temperature, remaining essentially constant at about 26%. However, the solubility of these elements in the γ phase decreased from 11% at 1154°C to 6% at 1077°C, as one would expect as the amount of γ' increases. The amount of Cr in the γ' phase decreased from 6% at 1154°C to 4% at 1077°C while the amount of Cr in the γ phase increased from 15% to 20% over the same temperature range. This result also is to be expected as Cr is known to partition strongly to the γ phase; therefore, as more γ' forms on cooling, the concentration of Cr in the γ phase must increase in order to accommodate that Cr rejected from the γ' phase.

For the other three elements (Mo, W, and Co) which were studied, only the Mo in the γ' phase and the Co in the γ phase changed more than 1 atom % over the temperature range observed. The Mo content of the γ' phase decreased 1% from 3.3% to 2.3% with decreasing temperature while the Co content of the γ phase increased 1.7% from 8.2% to 9.9% with decreasing temperature from 1154°C to 1077°C. It is interesting to note that Mo appears to partition toward the γ' at the temperature studied which is contrary to what would be expected from literature (ref. 2, 4, 5).

At the lowest temperature (1077°C) used in this study, the partitioning ratio is nearly one. If the decreasing trend of partitioning toward γ' were maintained with decreasing temperature values consistent with those of references 2, 4, and 5 might be expected at superalloy heat treating temperatures of 650 to 850°C where most studies have been performed.

Effect of Solution Temperature on Mechanical Properties

To determine what effect varying the solution temperature might have on tensile and stress rupture strength, As-HIP Rene' 95 specimens were solutioned at 1107°C or 1135°C, water quenched, aged 4 hours at 815°C followed by 24 hours aging at 650°C. The aged material was either tensile tested at room temperature or 650°C or stress rupture tested at 650°C with a stress of 1035 MPa. The results of the tests are listed in Table II.

The effects of solution temperature on ultimate tensile and yield strengths are shown in Figure 4. Both the yield strength at room temperature and 650°C and the room temperature ultimate strength decreased between 37 and 63 MPa when the solution temperature was decreased from 1135°C to 1107°C. But for the same change in solution temperature, the 650°C ultimate strength was not significantly changed. Tensile ductilities at both test temperatures were also not significantly changed.

Also shown in Figure 4 are data from reference 1 for which an oil quench from the solution temperature and a thicker section size were used. It can be seen that these conditions lead to a greater sensitivity to solution temperature. For example, for an 8°C increase in solution temperature, a marked increase occurred for both room temperature and 650°C

ultimate and yield strengths. The ultimate strength of the material in reference 1 particularly appears to be more affected by changes in solution temperature than the conditions tested in this investigation. The change of yield strength with change in solution temperature was similar for both this investigation and that of reference 1.

The stress rupture lives were also found to be sensitive to changing solution temperatures. Decreasing the solution temperature from 1135°C to 1107°C decreased the average stress rupture life (measured at 650°C and 1035 MPa) from about 473 to 100 hours (Table II). The decrease in rupture life with decreasing solution temperature was accompanied by a slight decrease in rupture ductility. Reference 1 reported that decreasing the solution temperature (for oil quenched material) from 1135 to 1127°C decreases the stress rupture life at 650°C with a stress of 1035 MPa from about 113 hours to about 80 hours. No change in rupture elongation was noted in reference 1, but the reductions in area decreased from 7 to 14% as the solution temperature decreased.

DISCUSSION

This investigation was performed to obtain additional information on the variation of the amount of γ' with solution temperatures near the γ' solvus temperature and the effect on mechanical properties of varying the solution temperature below the γ' solvus temperature. It was observed that as the temperature is decreased from the solvus temperature to about 50°C below the γ' solvus temperature the amount of γ' available for subsequent precipitation decreased at a rate of about 1% of the total amount available ($\frac{1}{2}$ vol. %) per degree Celcius.

Considering this high rate of change of amount of γ' with solution temperature together with the decreased mechanical properties associated with decreasing solution temperature both in this investigation and in the work of Bartos and Mathur (reference 1), it is apparent that the change in the amount of undissolved γ' is responsible, to a large extent, for the changes in mechanical properties observed. As the solution temperature is increased, more γ' is dissolved and subsequently precipitated as fine γ' during the heat treatment. As would be expected, the greater amount of fine γ' results in higher strength levels for the material. Therefore, if the highest strength levels are to be achieved with this material, the alloy should be solutioned just below the γ' solvus temperature. However, care must be taken not to exceed the solvus or grain growth will occur (reference 1). If the solution temperature decreases, strength levels decrease.

It is therefore recommended that the solution temperature be selected such that normal furnace variations will not cause the γ' solvus to be exceeded and extraordinary furnace practice should be applied to minimize the variation of temperatures during the processing to allow the greatest amount of γ' to be dissolved without chancing grain growth. For lower strength level applications, where the solution treatment is in the vicinity of the knee of the amount of γ' - temperature curve in figure 2, the importance of solution temperature selection and control should be greatly diminished.

Considering the compositions of both γ' and γ phases, it can be seen that the γ' composition is nearly constant with changing temperature.

Therefore, the precipitation of γ' with decreasing temperature is caused principally by the decreasing solubility of Al, Ti, and Nb, and the increasing solubility of Cr in the γ phase.

The material mechanically tested in this study had higher strength levels than those in reference 1. These higher strength levels are believed to be the result of the higher quench rate from the solution temperature resulting from water quenching rather than oil or hot salt quenching. It should be recognized that water quenching of turbine engine components would likely cause cracking and would not be used. In this study water quenching was used to achieve the highest strength levels from As-HIP Rene 95 in the expectation that the sensitivity of the strength to solution temperature would be exaggerated. The data suggests that, in fact, the strength of oil quenched As-HIP Rene 95 is more sensitive to solution temperature than that which was water quenched. No explanation for this effect can be offered at this time.

SUMMARY OF RESULTS

An investigation was performed on As-HIP Rene 95 to obtain additional information on the variation of volume fraction of γ' with solutioning temperatures near the solvus temperature. Also the resulting effects on the tensile and stress rupture strengths were evaluated. The principal results are as follows:

1. The amount of γ' increased at a rate of about 0.5% volume (1% of the total amount of γ') per degree Celsius decrease in temperature from the solvus temperature to about 50°C below the γ' solvus temperature.

2. The increase in the amount of γ' phase with decreasing solution temperature was primarily associated with the decreasing solubility of Al+Ti+Nb and increasing solubility of Cr in the γ phase. For example, the solubility of Al+Ti+Nb in the γ phase decreased from about 11 atom % at 1154°C to about 6% at 1077°C and the solubility of Cr in the γ phase increased from about 15% to 20% over the same temperature range.
3. For Rene' 95 which was aged 4 hours at 915°C followed by 24 hours at 650°C after being water quenched from solution temperature of 1107°C and 1135°C the following changes in strengths were observed: (a) The room temperature and 650°C yield strengths and the room temperature ultimate strength were about 50 MPa greater for the higher solution temperature. The 650°C yield strength appeared to be slightly lower for the higher solution temperature. (b) The average stress rupture life at 650°C and a stress of 1035 MPa was 100 hours for the 1107°C solution temperature and 473 hours for the 1135°C solution temperature.
4. The amount of γ' phase available for subsequent precipitation and strengthening when Rene 95 is solutioned near the γ' solvus temperature is very sensitive to the solution temperature. It is therefore recommended that extraordinary temperature control be used when solutioning As-HIP Rene' 95 so that the maximum strength can be achieved by solutioning at the maximum nominal temperature which will not exceed the γ' solvus temperature and cause grain growth.

REFERENCES

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3. E. H. Van Der Molen, J. M. Oblak, and O. H. Kriege: Metall. Trans., 1971, vol. 2, no. 6, p. 1627.
4. R. L. Dreshfield, and J. F. Wallace: Metall. Trans. 1974, vol. 5, no. 1, p. 71.
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Major Elements, Wt. %

	C	Cr	Mo	Co	Ti	Al	Nb	W	Zr	B
Max.	.09	14	3.7	9	2.7	3.7	3.7	3.7	.07	.015
Min.	.04	12	3.3	7	2.3	3.3	3.3	3.3	.03	.006

Other Elements

	Mn	Si	P	S	Ta	Fe	O ₂	N ₂	H ₂
Max.	.15	.2	.015	.015	.2	.5	150ppm	50ppm	10ppm

TABLE I Composition Range of PM Rene 95
(ref. 1)

Solution Temperature °C	Test Temperature °C	Ultimate Strength, MPa	.2% Yield Strength, MPa	Elongation %	Reduction in area %
1135	Room Temperature	1785	1415	13	16
1135	Room Temperature	1775	1390	10	13
1107	Room Temperature	1705	1380	12	12
1107	Room Temperature	1730	1350	10	14
1135	650	1530	1290	6	8
1135	650	1510	1290	10	8
1107	650	1530	1245	7	8
1107	650	1540	1215	9	9

A. Tensile^a

Solution Temperature °C	Life Hrs.	Elongation %	Reduction in area, %	Note
1107	131	1	3	a
	69	1	4	a
1135	398	4	4	a
	549	2	6	a
1127	89	5	15	b
	69	4	13	b
1135	109	5	7	b
	117	4	7	b

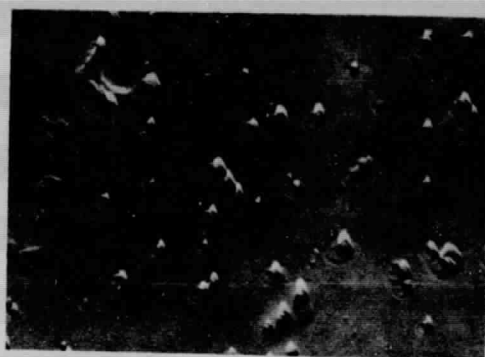
B. Stress Rupture 650°C/1034 MPa

- a. water quench + 815°C/4hrs + 650°C/24hrs
b. oil quench + 870°C/1hr + 650°C/24hrs - Bartos & Mathur, ref. 1

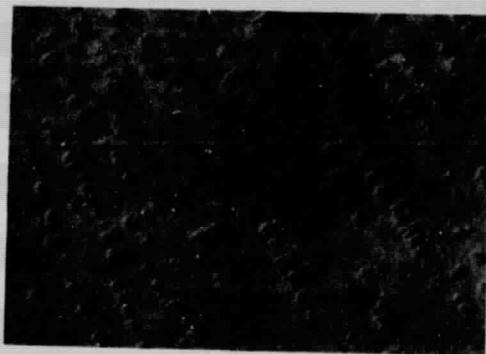
TABLE 11 Effect of Solution Temperature on Mechanical Properties



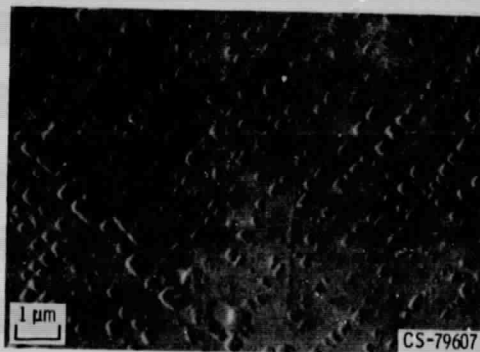
(a) 2 HOURS AT 1190°C PLUS 6 HOURS AT 1171°C.



(b) 2 HOURS AT 1190°C PLUS 6 HOURS AT 1154°C.



(c) 2 HOURS AT 1190°C PLUS 6 HOURS AT 1113°C.



(d) 2 HOURS AT 1190°C PLUS 6 HOURS AT 1077°C.

Figure 1. - Microstructures of Rene 95 solutioned near γ' solvus.

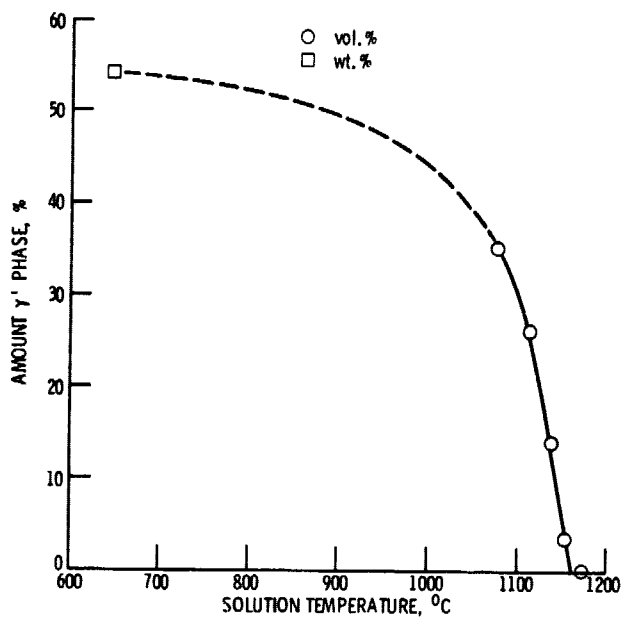


Figure 2. - Effect of solution temperature on the amount of γ' phase in PM Rene' 95.

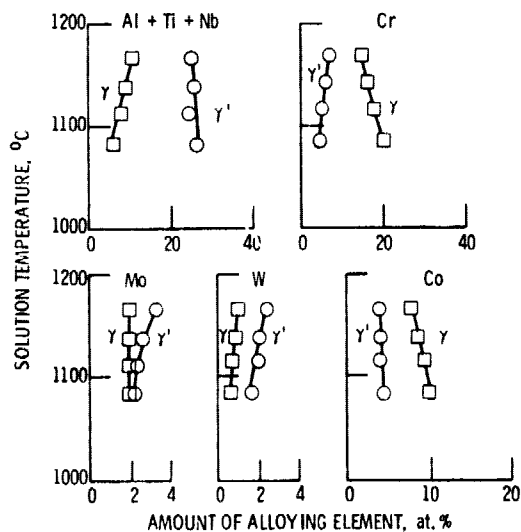


Figure 3. - Constitution diagram for Rene' 95.

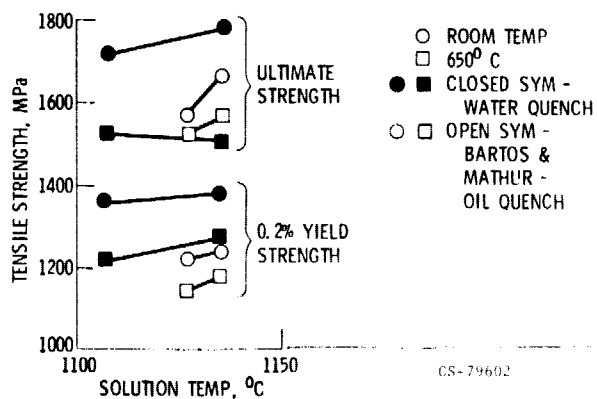


Figure 4. - Effect of solution temperature on tensile strength of As-HIP Rene' 95.